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Microstructure tailoring for property improvements by grain boundary engineering

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Abstract

Grain boundary engineering (GBE) was employed to improve materials properties such as corrosion resistance and strength by optimizing the grain boundary character distribution. Two high-temperature alloys, designated Incoloy 800H and Inconel 617 were selected in this study due to their potential applications for the Generation IV nuclear power systems. The GBE treatments on the alloys 800H and 617 were accomplished by a series of thermomechanical processing. The effect of the GBE treatments on the corrosion resistance and mechanical properties of the materials were evaluated using supercritical water exposure tests, cyclic oxidation tests, impact tests, and tensile tests. The microstructures of the tested samples were analyzed by means of optical microscopy, scanning electron microscopy, energy-dispersive X-ray spectroscopy, electron backscatter diffraction, X-ray photoelectron spectroscopy, and grazing incidence X-ray diffraction. The results indicate that the GBE treatments greatly mitigated the oxide exfoliation of the alloy 800H and reduced the oxidation rate of the alloy 617. The GBE treatment also greatly enhanced the strength of alloy 800H at room temperature (e.g. impact tests) and high-temperatures (e.g. tensile tests after neutron irradiation), but did not significantly impair the material's ductility.

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1. Introduction

Grain boundaries are common defects existing in crystalline materials, and play a major factor in determining the physical, mechanical, electrical, and chemical properties of crystalline materials [1]. Based on the misorientation between adjacent grains, grain boundaries can be categorized as low-angle boundaries (LABs) with a misorientation angle generally less than 15° and high-angle boundaries (HABs). Using the concept of the coincidence site lattice (CSL), they also can be categorized as $\Sigma 1$ (maximum misorientation angle 15°) boundaries corresponding to the LABs [2], and low-Σ CSL boundaries (CSLBs, $3 \leqslant \Sigma \leqslant 29$) and random boundaries corresponding to the HABs. The Σ is a value defined as the reciprocal density of coincident sites at the grain boundary between two adjoining grains.

Compared to random boundaries, low-Σ CSLBs have many special properties such as low boundary energy, less susceptibility to impurity or solute segregation, and greater resistance to grain boundary sliding and intergranular degradation [2]. Due to the special properties of the low-Σ CSLBs, grain boundary engineering (GBE) was proposed as an approach to control the properties of polycrystalline metals by tuning grain boundary character distribution (GBCD) to obtain a high fraction of low-Σ CSLBs and interrupt the connectivity of random boundaries. GBE has been used to improve the properties of polycrystalline metals such as strength [3], creep [4], weldability [5], and stress corrosion cracking [6]. Among the low-Σ CSLBs, the contribution of $\Sigma 3$ boundaries to the property improvements has

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been found to be the most significant [7,8]. This is because the energy of $\Sigma 3$ boundaries is extremely low, typically about 1/50 of a random boundary [2]. Detailed information about the CSL model and CSL effect on GBE can be found in Ref. [2].

Successful GBE by means of thermomechanical processing has been applied to face-centered-cubic (FCC) materials with low stacking-fault-energies. Thermomechanical processing is a combination of deformation and annealing to produce annealing twins. The applied thermomechanical processing can generally be categorized as recrystallization, featuring a high level of cold work followed by a short high temperature anneal, and strain annealing, featuring a low level of cold work followed by a long medium/high temperature anneal [9].

Incoloy alloy 800H (UNS N08810, Fe–31Ni–20Cr) and Inconel alloy 617 (UNS N06617, Ni–22Cr–13Co–9Mo) were studied in this work due to their potential applications in the Generation IV Nuclear Power Systems [10]. The detailed specifications of these two alloys regarding the physical and mechanical properties could be found in Ref. [11]. Both of these alloys are solid-solution-strengthened alloys with additional strengthening by precipitation of titanium nitrides and carbides such as MC (rich in Ti), $M_{23}C_6$ (rich in Cr), and M_6C (rich in Ni and Mo in alloy 617). Titanium nitrides are stable at all temperatures below the melting point and are therefore unaffected by heat treatment [11]. Furthermore, the γ'-phase such as $Ni_3(Al,Ti)$ was observed in alloy 617 at temperatures between 650 and 760 °C [11]. Both of these two alloys have good creep resistance. However, extensive oxide exfoliation was observed on alloy 800H following exposure to supercritical water (SCW) [12]. The corrosion resistance of alloy 617 was found inappropriate for very high temperature reactor (VHTR) application [13]. This study was performed primarily to improve the corrosion resistance of these alloys by means of GBE with limited testing to investigate the effect of GBE on radiation response.

SCW is an attractive superfluid existing at temperatures and pressures above the critical point of water at 374 °C and 22.1 MPa. It has been used in modern power plants to improve thermal efficiency and reduce the release of deleterious gases such as carbon dioxide, nitrogen oxides, and sulfur oxides. Due to these advantages, SCW has been proposed as a coolant for Generation IV nuclear power plants [10]. Preliminary SCW exposure tests indicated that GBE could be an effective approach to mitigate oxide exfoliation of alloy 800H at 500 °C [12]. This paper presents the effect of GBE on the corrosion resistance and strength of alloys 800H and 617.

2. Experiments

The materials used in this study were commercial alloys Incoloy 800H and Inconel 617 and the chemical compositions provided with the materials are listed in Table 1. Both of the as-received alloys were annealed at ~1177 °C for a time commensurate with section size followed by water quenching. Thermomechanical processing was performed on the as-received samples cut from the alloys by a series of a low level of cold work followed by a high temperature anneal and water quenching. Based on the previous experience [14,15], a series of thermomechanical processing with a ~6% thickness reduction followed by annealing at 1050 °C for 90 min for alloy 800H and a ~5% thickness reduction followed by annealing at 1100 °C for 90 min for alloy 617 were employed for the GBE treatments.

2.1. Supercritical water (SCW) exposure tests

The as-received and the GBE-treated alloys 800H and 617 were subjected to SCW exposure tests. Rectangular samples (31.7 mm × 12.7 mm) with a thickness of ~1 mm were cut from the as-received and the GBE-treated materials then polished down to a 1 μm surface finish and ultrasonically cleaned prior to the exposure. The SCW was maintained at 500 or 600 °C and ~25 MPa with a test section inlet dissolved oxygen content of ~25 ppb and a flow rate of ~1 m/s. The applied heating and cooling rates were ~1 °C/min.

2.2. Cyclic oxidation tests

Cyclic oxidation testing is a key method to aid material selection and to predict service lifetime of components. The as-received and the GBE-treated alloys 800H and 617 samples were subjected to cyclic oxidation testing in air at a variety of temperatures such as 500 and 850 °C for the 800H samples and 850 and 1000 °C for the 617 samples. Two as-received and two GBE-treated samples were tested at the same time for each alloy at a designated cyclic exposure temperature. Each cycle was composed of putting samples in a furnace at designated temperatures for a heating period of one day and followed by pulling samples out for an air cooling to room temperature for about 15 min to measure the weight changes. An electronic balance with a sensitivity of 0.1 mg was employed.

Table 1
Chemical composition (wt%) of Incoloy 800H and Inconel 617

Alloy	Fe	Cr	Ni	Co	Mo	Mn	Cu	Si	Ti	Al	Others
800H	45.26	20.42	31.59	–	–	.76	.42	.13	.57	.50	C, S, P
617	1.47	22.05	52.32	12.69	9.35	.27	.11	.15	.38	1.07	C, S, B